

THE PENNSYLVANIA STATE UNIVERSITY
SCHREYER HONORS COLLEGE

DEPARTMENT OF CHEMICAL ENGINEERING

Analyzing the Selectivity of Commercial Cation Exchange Membrane by Electrodeposition of
Polyethyleneimine (PEI)

MATTHEW FISHER
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A thesis
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in Chemical Engineering
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Reviewed and approved* by the following:

Bruce Logan
Professor of Civil and Environmental Engineering
Thesis Supervisor

Ali Borhan
Professor of Chemical Engineering
Honors Adviser

* Electronic approvals are on file.

Abstract

The continual growth of technology in today's world is resulting in an increase in demand for the rare earth elements (REEs) that make up certain microchips. Applications of these microchips range from devices such as mobile phones to ones found in wind turbines. Certain locations within the United States have proven to house enough resources for REEs, however, the reserves of REEs are limited. The objective of this thesis is to study one of the ways REEs can be separated and extracted from mining wastewater found in some of these reserves. One of the current ways of separating ions from water is using processes with ion exchange membranes. The two types of ion exchange membranes are cation exchange membranes (CEM) and anion exchange membranes (AEM). In this study I analyzed the permselectivity of commercial cation exchange membranes for monovalent, divalent, and trivalent ions. Trivalent ions are specifically important since the relevant REEs share the same property. The selectivity analysis was performed for the salts NaCl, CaCl₂, MgCl₂, and CeCl₃. The first analysis was done using pristine CEM to observe the behavior of ions through an uncoated membrane surface. Then, CEM were coated with the polymer polyethyleneimine (PEI) through means of electrodeposition to be used for the next permselectivity analysis runs. The goal of depositing PEI on the membrane surface was to create enough repulsive forces between the coated layer to successfully reject trivalent ions. Permselectivity data for the coated membrane showed lower values for CeCl₃ across the membrane, proving that the technique could efficiently reject the trivalent ions and potentially be a viable method for separating REEs from wastewater.

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4 Introduction

Rare earth elements (REEs) are becoming extremely prominent in the world's pursuit of technological advancement. Scientists are finding usages for these elements in microchips for systems ranging from cell phones to wind turbines. The rare earth elements include the lanthanide series elements (La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu) plus Sc and Y.¹ Although they are labeled with the description of "rare", this does not imply they are hard to find. Instead, the rareness of these elements pertains to how difficult they are to extract into a usable form.² In other words, there are enough resources of REEs. However, the reserves of REEs are limited.

Most of these elements have been found in abundant amounts in wastewater from many different operations. However, when considering which have a low natural abundance and high demand for production, Nd, Y, Eu, Tb, and Dy are considered the most economically critical rare earth elements.³ Most mining fields with reserves of rare elements are found in China, but recently, there has been an investigation into the mines of the United States to see if there is an opportunity to source them domestically. With this, American scientists are tasked with finding efficient ways to separate these elements from the water and preserve them for their further applications.

Most rare earth elements are recovered from bastnaesite ores which are limited to the western coast of the United States, followed by a secondary source of monazite deposits which are not present there at all. Another source that was utilized in Poland as a resource for the elements came from dumps of phosphogypsum. Phosphogypsum is an acid byproduct from the phosphate mining industry which is primarily made up of calcium sulfate dihydrate.⁵ Sixty to 80% of the rare earth elements present in phosphate rock may be partitioned into the

phosphogypsum (dihydrate or hemihydrate) during the production of phosphoric acid.⁵ In the United States, phosphogypsum is present in both central and northern Florida as well as Idaho.

Table 1. Content (mg kg⁻¹) of selected rare earth elements in dihydrate phosphogypsum produced from Idaho phosphate rock and literature values for terrestrial materials.⁵

Element (Z) ^b	Idaho		Shale ^c	Soil ^d
	Mean	Range		
La (57)	81	69–90	40	37
Ce (58)	36	31–45	70	49
Nd (60)	36	30–46	30	46
Sm (62)	5.4	4.7–6.3	7	6.1
Eu (63)	1.2	1.1–1.4	1.4	1.9
Tb (65)	1	0.8–1.2	1	0.63
Yb (70)	2.8	2.6–3.5	3.5	2.3
Lu (71)	0.41	0.36–0.51	0.6	0.34

There are currently many practices used in industry for separating rare earth elements from phosphogypsum and other wastes. The REEs have similar chemical properties, which translate into separation and purification difficulties.⁶ Some examples of separation strategies include different adsorption techniques and hydro-, pyro- or electro-chemical processes. Another prominent technique used in industry is using selective membranes to promote extraction and rejection of the element's ions. Specific methods have been used for the separation and recovery of REEs using membranes, including supported liquid membranes (SLMS), polymer inclusion membranes (PIMS), ion imprinted membranes (IIMS), nanocomposite membranes, and ion exchange membranes. Cation exchange membranes (CEMs) and anion exchange membranes (AEMs) are two types of ion exchange membranes (IEMs). These membranes have been extensively studied to determine the permselectivity of monovalent ions. Their application in the separation of rare earth elements comes with testing the permselectivity that they have for trivalent ions.

The objective of this project was to test cation exchange membranes and their selectivity for ions that can be representative of those within rare earth elements. Cation exchange membranes are commonly used as selective separators in diverse water purification processes, such as electrodialysis and diffusion dialysis.¹¹ These membranes alone contain fixed negatively charged groups such as sulfonic acid and carboxylic acid groups, allowing positively charged ions in solution to pass through. With the goal of enhancing the selectivity of these membranes, several surfactants and polymers have been introduced into such systems. One polymer that has been thoroughly studied is polyethyleneimine. The goal of this specific experiment was to utilize a polyethyleneimine coating to enhance the rejection of trivalent cations.

5 Materials and Methods

5.1 Polyethyleneimine

Polyethyleneimine, also known as PEI, is a positively charged polyelectrolyte where the degree of protonation depends on pH value.¹² Because of this property of the molecule, it could be used and coated on the surface of ion exchange membranes. When the PEI is dissolved in the acid, the amine groups in the PEI become a source of positive charges. By coating the polyelectrolyte on the surface of membranes, a layer of positive charge forms on the surface of CEM, allowing the CEM to reject positive ions due to repulsive forces between the ion and the positive layer (PEI layer) on the surface of modified CEM. As shown in Figure 1, PEI has three types of amine groups, including primary, secondary, and tertiary amine groups, making it an ideal polyelectrolyte with a potential for protonation in the presence of acid. The protonated positions are marked by red boxes.

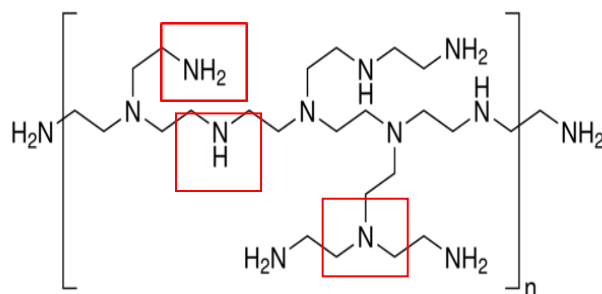


Figure 1. Branched PEI structure.

One thing to consider when adding PEI to cation exchange membranes is what concentrations of the polymer to use. Also, the acid used to dissolve the PEI will influence the degree of protonation it will experience. In this experiment, 0.1 molar hydrogen sulfate (H_2SO_4) was used. Because its pH is between 1 and 2, the degree of protonation was over 95%. Concentrations of 1, 3, and 5 grams per liter PEI- H_2SO_4 solutions were made so the effects of different PEI amounts on the surface of the membrane could be analyzed. The PEI was adsorbed on the membrane structure by electrodeposition.

5.2 Electrodeposition

There are multiple methods for coating ion exchange membranes, but electrodeposition was chosen for this process. Electrodeposition is typically used to coat metals with a thin layer of another metal. In this experiment, however, the PEI from the prepared PEI- H_2SO_4 solutions was deposited onto a cation exchange membrane. A graphic of the electrodeposition process and a picture of the experimental setup are depicted in Figure 2 below.

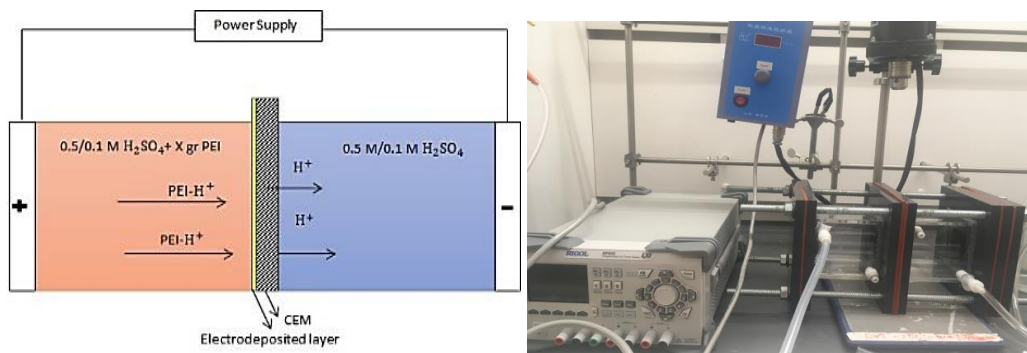


Figure 2. Electrodeposition process and experimental setup.

As seen on the left-hand side of Figure 2, the H_2SO_4 -PEI solution was placed in one cell and a pure H_2SO_4 solution filled the other. As previously mentioned, 0.1 molar H_2SO_4 was utilized for this specific experiment. In between the two cells was the cation exchange membrane, and potential was applied on both ends of the system. The CEM has negative charges (sulfonic acid groups) and the PEI which was protonated by the acid has positive sites. So, PEI molecules diffuse towards the membrane under a current field effect and then adsorb or penetrate the membrane pores. The protons can pass through, but PEI molecules are not able to pass due to their large size. As a result, one side of the membrane becomes coated with the PEI polymer.

In addition to the concentration of PEI used for the electrodeposition process, other parameters were varied to analyze their effects in coating the membranes. The three other parameters that were changed were time, potential, and current. The times used were 1, 3, and 5 hours. 10, 25, and 30 volts were used for the potential. Finally, the current was varied between 1, 2, and 2.5 amperes. Following many trials to find the parameters that maximize the deposition of PEI onto the membranes, the next step was to compare these coated membranes to pristine cation exchange membranes through selectivity analysis.

5.3 Selectivity Analysis

In ion-exchange membranes, the permselectivity is related to the transport of charges by the counterions. The counter ion for a CEM is a cation (in this case Na^{+1} , Mg^{+2} , Ca^{+2} , and Ce^{+3}). To determine the permselectivity of the membranes, electrochemical potential measurements were taken in a two-cell system separated by a CEM. The measurements were taken by an instrument known as a potentiostat (BioLogic SP-300, US). A schematic and image of the experimental setup are shown below in Figure 3.

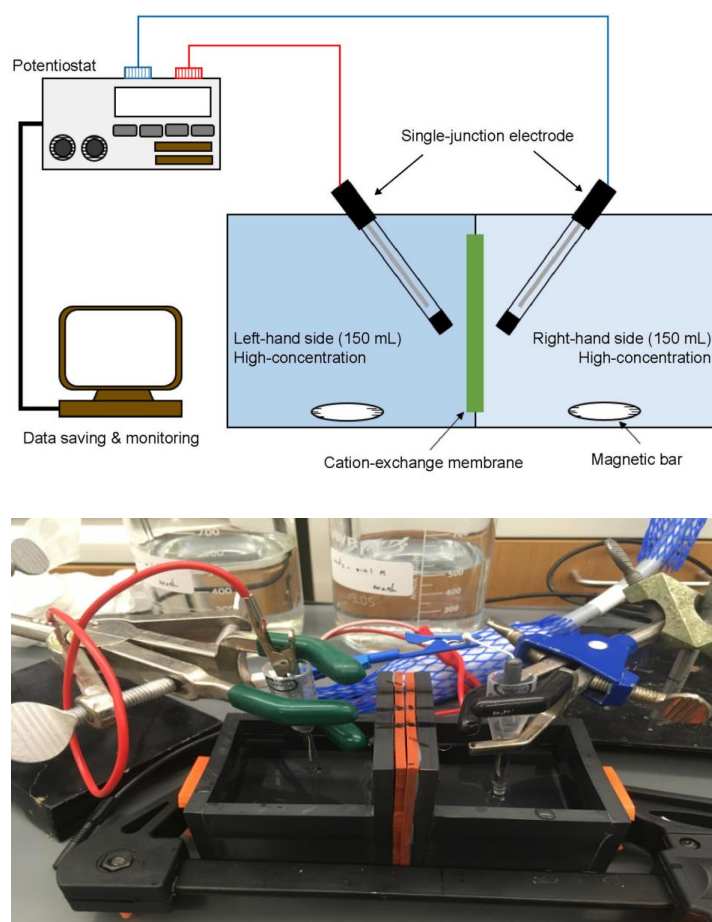


Figure 3. Permselectivity cell and experimental setup.¹¹

As seen in Figure 3, the two-cell setup was similar to that of the electrodeposition system. At first, 0.01 and 0.1 molal solutions were made for each of the four salts. The four salts used were sodium chloride, magnesium chloride, calcium chloride, and cerium chloride. These four salts gave us the ability to measure the selectivity of monovalent, divalent, and trivalent ions through the CEMs. Upon completing the first set of experiments with the chosen concentrations, it was determined that the solutions needed to be more concentrated to produce accurate results. Therefore, new solutions were made with the concentration of 0.05 molal (m) as the diluted solution and 0.5 molal for the concentrated solution.

The new concentrations were deemed to be a more accurate representation of the system's behavior. The first run through each salt involved two runs with each and the use of a pristine CEM between the two cells. Data were collected through the reference electrodes (Ag/AgCl) by the potentiostat for five minutes to provide a series of open-circuit voltage (OCV) values. OCV is simply the difference in electrical potential between the two cells when there is no current in the circuit.

There are several methods for calculating permselectivity through such an experiment. For this specific project, the Bromley model and Nernst equation were chosen. The first step in the Bromley model involves calculating the activity coefficient for both the concentrated and diluted solutions. This is done using

$$\log \gamma_{\pm}^{\frac{1}{|Z_+Z_-|}} = \frac{-0.511\sqrt{I}}{1 + \sqrt{I}} + \frac{(0.06 + 0.6B)I}{\left(1 + \frac{1.5}{|Z_-Z_+|}I\right)} + \frac{BI}{|Z_-Z_+|} \quad (1)$$

where γ is the activity coefficient, Z is the charge number on each of the ions, I is the ionic strength, and B is a constant specific to each salt. Once the activity coefficient was calculated, the value, along with the average OCV value collected from the potentiostat, were applied to the Nernst equation. The form of the Nernst equation used is shown in Equations 2 and 3,

$$\alpha = \frac{OCV_{experimental}}{OCV_{ideal}} \quad (2)$$

$$OCV_{ideal} = \frac{RT}{zF} \ln \left(\frac{m_c \gamma_c}{m_d \gamma_d} \right) \quad (3)$$

where α is the permselectivity, R is the gas constant (8.314 J/mol/K), T is the temperature (K), z is the charge on the cation, F is Faraday's constant (96485 C/mol), m_c is the molality of the concentrated solution, γ_c is the activity coefficient of the concentrated solution, m_d is the molality of the diluted solution, and γ_d is the activity coefficient for the diluted solution. By completing these calculations for two separate trials, an average selectivity was found, and a standard deviation was calculated. The same process was repeated for each of the four salts but this time with a PEI-coated membrane. For each salt, one trial was completed with a membrane that was coated through electrodeposition at 30 volts for five hours.

6 Results and Discussion

The first step in the data analysis involved using the Bromley model (Equation 1) to calculate the activity coefficient for each salt at the chosen concentrations. For each, the concentrations and charge of the cations determined the ionic strength which in turn altered the results of the activity coefficient value for each. The results of the activity coefficient calculations for the four salts are depicted in Figure 4 below.

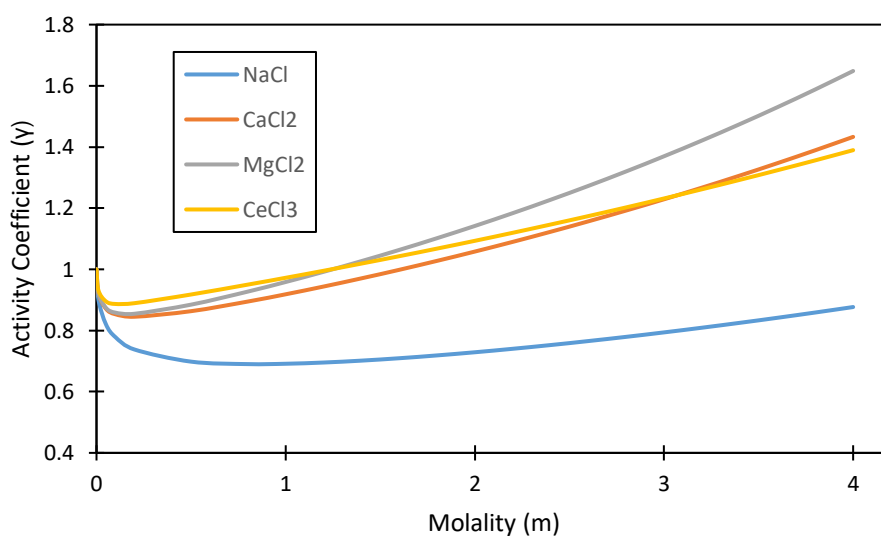


Figure 4. Activity coefficient versus concentration for chosen salts.

As seen in Figure 4, the activity coefficients all started at 1 when considering pure distilled water (ideal solution). As the concentration increased, there was a clear discrepancy between the monovalent, divalent, and trivalent ion compounds. This pattern follows the same relationship between the compounds and their corresponding B values. The lowest activity coefficients were seen in the monovalent ion salt (NaCl) which has a B value of 0.0574 kg/mol. Looking all the way to the right side of the plot, as concentration gets higher, the CeCl₃ ends up having the next lowest activity coefficients. For lower concentrations, however, the CeCl₃

activity is above that of CaCl_2 . Based on the B values of CeCl_3 and CaCl_2 , which are 0.0815 kg/mol and 0.0948 kg/mol respectively, it can be inferred that the ionic strength of the compounds dominates at lower concentrations, and the B value dominates at higher ones. Furthermore, looking at the second divalent ion compound (MgCl_2), which has a B value of 0.1129, the same relationship is seen with CeCl_3 . This again proves that a higher B value will have more domination over ionic strength when considering the compound's activity. These calculated activity coefficients played an important role in analyzing the permselectivity of each salt in the next stage of the experiment.

The permselectivity analysis was performed using the two-cell system shown in Figure 3 and a potentiostat. The potentiostat measures OCV at a set time interval and duration. For these experiments, measurements were taken every second for a duration of five minutes. This was repeated until steady-state results were produced, showing that the system was stabilized. An example graph of the data produced is shown below in Figure 5.

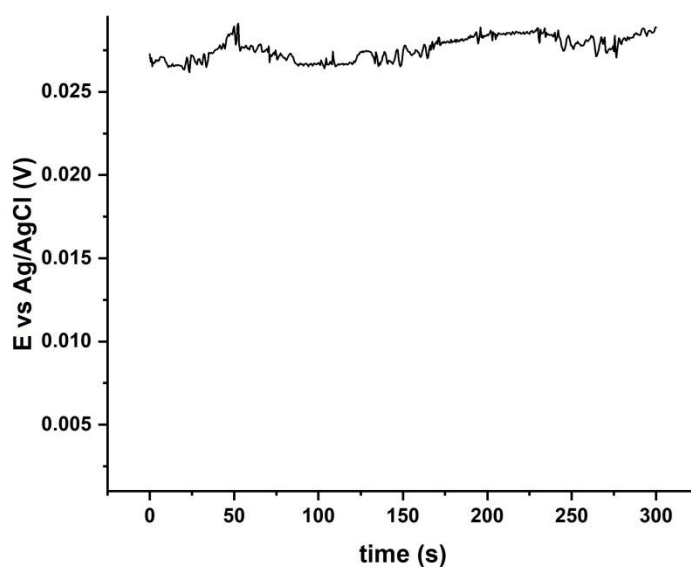


Figure 5. Example OCV results from potentiostat for CaCl_2 .

The first set of runs was with the pristine cation exchange membranes. Once stable OCV data for two separate runs was collected for each of the four salts, the permselectivity was calculated with both values using Equations 2 and 3. Then, the average of these results was taken and used as the average permselectivity of the pristine membrane for the specific salt. For NaCl, the OCV values were measured to be 0.053 V and 0.055 V. These values yielded an average permselectivity of 98.2%. The OCV values collected for two runs of CaCl₂ analysis were 0.0261 V and 0.02673 V. A permselectivity of 89.7% was calculated from these results. The second divalent ion compound (MgCl₂) produced OCV values of 0.028 V and 0.0285 V, yielding a permselectivity of 95.0%. Lastly, CeCl₃ was measured to have OCV values of 0.0175 V and 0.0203 V. The average of the OCV values yielded a permselectivity of 94.8% for the trivalent ion compound. A standard deviation was calculated for the two OCV results of each compound to provide a range of errors in the data. The summary of the permselectivity results for each of the four salts is shown below in Figure 6.

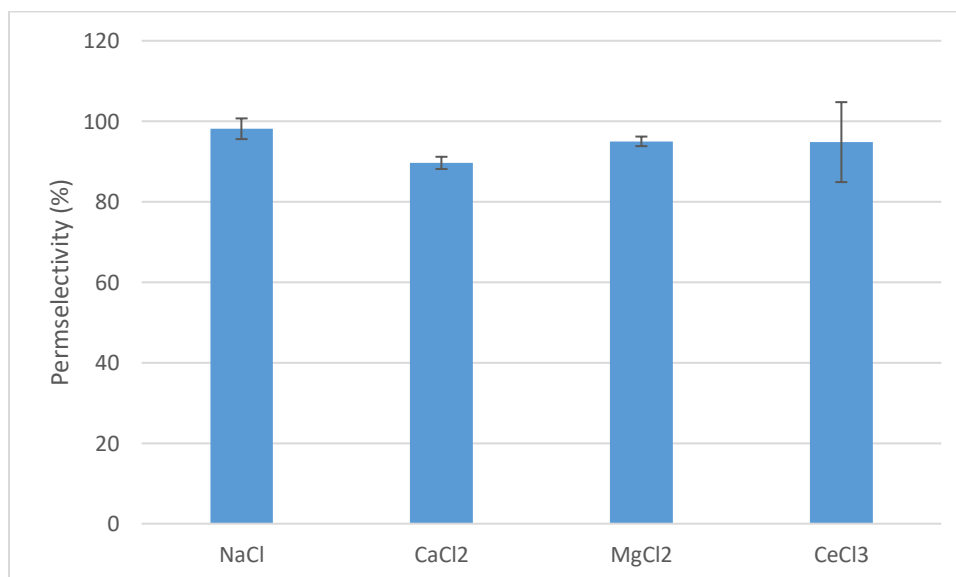


Figure 6. Permselectivity results for pristine membrane trials.

As seen in Figure 6, the permselectivity for NaCl was the highest, followed by MgCl₂, CeCl₃, then CaCl₂. This shows that the pristine membranes were most selective for monovalent ions which was expected due to the higher affinity of Na ions to functional groups. The discrepancy between the two divalent ions could be explained by magnesium's stronger ionic strength which makes it harder to be separated. As seen in the data, especially for CeCl₃, there may have been some errors within the results. This error could be attributed to simple experimental errors such as temperature and measurement.

As mentioned, further permselectivity analysis was performed using membranes coated with PEI. Through electrodeposition, the membranes were coated with 3 grams of PEI, dissolved in 0.1 M sulfuric acid, at 30 volts for a duration of 5 hours. The goal of these trials was to see how effective the coated membranes were in separating the trivalent ion of CeCl₃ and how the results compared to those of the uncoated membranes. For NaCl, the OCV values obtained were 0.0553 V and 0.05538 V. The average of these values yielded a permselectivity of around 100%. The CaCl₂ runs gave OCV values of 0.0265 V and 0.0269 V. The associated permselectivity was calculated to be 95.3%. The MgCl₂ samples provided OCV results of 0.02821 V and 0.02861 V. The permselectivity of MgCl₂ was determined to be 97.8%. Lastly, CeCl₃ was tested and yielded OCV results of 0.0012 V and 0.0025 V, giving a permselectivity of 54.6%. A summary of the coated membrane results along with a direct comparison to the uncoated membrane results is shown below in Figure 7.

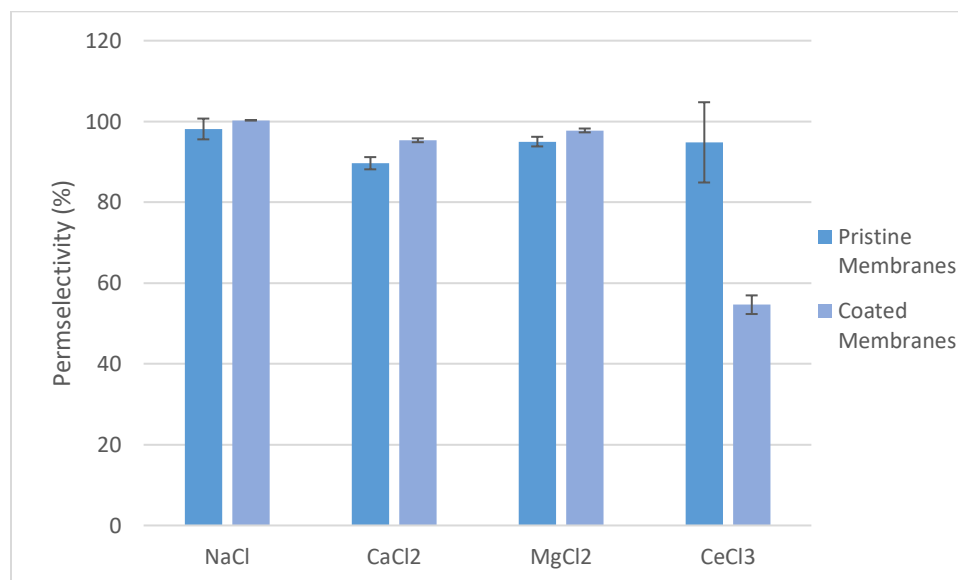


Figure 7. Permselectivity results for coated membrane trials in comparison to uncoated membrane results.

As seen in Figure 7, the coated membranes had the highest permselectivity for NaCl, followed by MgCl₂, CaCl₂, the CeCl₃. These results differ from those of the uncoated membranes as CeCl₃ had a much lower permselectivity than the rest of the salts which increased. The increase in permselectivity of the monovalent and divalent ions seems to be due to protons attached to the CEM during the modification process with PEI and sulfuric acid. Also, these results suggest that the electrodeposition method does not create enough repulsive forces between the coated layer and the mono/divalent ions, allowing them to pass through. The decrease in permselectivity for CeCl₃ validated the electrodeposition process and its ability to create enough repulsive forces between the deposited layer of PEI and trivalent ions. The low permselectivity proves that the trivalent ions were rejected efficiently. Again, some error was

found in the results, but it was not significant enough to invalidate the behavior and efficiency of the system.

7 Conclusions

This thesis provided raw experimental data for the selectivity of commercial cation exchange membranes by the electrodeposition of polyethyleneimine (PEI). The goal was to analyze the permselectivity of the membranes for monovalent, divalent, and trivalent ions with specific interest in the results for the trivalent ions due to their relevancy to rare earth element characteristics. The monovalent ion selected was sodium in the salt NaCl. The two divalent ion compounds were CaCl₂ and MgCl₂. The trivalent ion compound which was the most significant species in the study was CeCl₃.

The permselectivity of each ion was first tested with a pristine CEM to observe the behavior of the uncoated membrane. The open circuit voltage of each salt was first measured by a potentiostat and the obtained values were then used to calculate the permselectivity of each. The uncoated membranes are designed for monovalent ions but are not very selective between mono and multivalent cations. This was evident in the experimental data as the permselectivity for NaCl was around 98%, higher than any of the other cations. This phenomenon could be explained by the higher affinity of Na ions to functional groups (sulfonic acid groups in the CEMs). The next highest permselectivity was that of MgCl₂ (95.0%), followed by CeCl₃ (94.8%), then CaCl₂ (89.7%).

Following the permselectivity analysis of the pristine membranes, further analysis was done using membranes coated with PEI. The coating method used was electrodeposition and the chosen parameters were 3 grams of PEI, a potential of 30 volts, and a time duration of 5 hours. Following the electrodeposition of PEI, the four salts were analyzed by using the same methods as the pristine membranes. The permselectivity of NaCl, CaCl₂, and MgCl₂ were all larger compared to their trials with the uncoated membranes. The permselectivities of NaCl, CaCl₂, and

MgCl₂ through the coated membranes were 100%, 95.3%, and 97.8% respectively. The increase seen in permselectivity may be explained by the attachment of protons to the CEM during the electrodeposition process. The results prove that the electrodeposition method did not generate enough repulsive forces to reject mono or divalent ions. The CeCl₃ showed the opposite behavior as its permselectivity (54.6%) was much lower through the coated membrane compared to the uncoated. This trend suggests that the electrodeposition successfully created enough repulsion forces between the layer of PEI and the trivalent ions to efficiently reject them. Therefore, the overall process utilized in this experiment seems to be a viable option for the separation of trivalent REEs from wastewater. Further experiments could potentially adjust some parameters of the systems to increase the final permselectivity of ions and reduce any error associated with them.

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MATTHEW FISHER

mlf5648@psu.edu

Skills

- Microsoft Office Suite (Excel, PowerPoint, Word)
- Mathematica programming
- Chemistry Lab experience
- Undergraduate Research
- Multidisciplinary Team Collaboration
- Polymath

Experience

Penn State University

Dr. Bruce Logan's Research Lab

08/2022 - Current

- Performed research with a focus on selectivity analysis of commercial cation exchange membranes. Working on an honors thesis project for Schreyer Honors College.

PPL Electric Utilities | Hazleton, PA

Engineering Intern

05/2023 - 08/2023

- Gained experience in the Engineering Reliability department. Responsibilities included proactive circuit analysis and project cost/benefit analysis. Worked extensively on electrical load forecasting project.

Susquehanna Nuclear LLC (Talen Energy)

Engineering Intern

06/2022 - 08/2022

- Contributed to the Equipment Reliability department. Gained introductory experience in nuclear power operation and industry. Responsibilities included project prioritizing and ranking.

Penn State University

Dr. Lauren Greenlee's Research Lab

12/2021 - 05/2022

- Worked to sustainably mine rare earth elements from fertilizer byproducts (work with electrodeposition and selectivity analysis)

Education and Training

The Pennsylvania State University | University Park, PA

Schreyer Honors College

Bachelor of Science in Chemical Engineering

Expected in 12/2023

- Relevant Courses: Engineering Design Processes, Physical and Organic Chemistry Labs, Chemical Engineering Comp Tools, Thermodynamics, Process Heat Transfer, Mass Transfer and Separations, Chemical Plant Design

Leadership And Involvement

- **Sustainable Energy Corps**, Built mitigation plans for local emission points, founding member, 2022
- **THON**, Committee member, Set up a donor drive and gained THON experience, 2020-2021
- **AIChE** – American Institute of Chemical Engineers, Penn State Chapter, Member, 2020-Present